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Indian Standard

SPECIFICATION FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

(First Revision)

UDC 661.844.43 : 622.221.212

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *September* 1982

AMENDMENT NO. 1 JUNE 2005
TO
IS 4396 : 1981 SPECIFICATION FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS
(First Revision)

(*Page* 4, *clause* **2.2**) — Insert the following subclause after the clause **2:**

2.2.1 For defence purpose the sieving requirements are as follows:

ieving Requirements	*Limit*
) Size 250 micrometre Retained on 250 micrometre IS sieve	Nil
) Size 250/125 micrometre	Nil
i) Retained on 250 micrometre IS sieve	Nil
ii) Retained on 125 micrometre IS sieve, percent by mass, *Min*	90
) Size 150 micrometre Retained on 150 micrometre IS sieve	Nil
) Size 125 micrometre Retained on 125 micrometre IS sieve	Nil
i Size 250 micrometre A	
i) Retained on 250 micrometre IS sieve	Nil
ii) Retained on 150 micrometre IS sieve, percent by mass, *Max*	50
Size 125 micrometre A	
i) Retained on 180 micrometre IS sieve	Nil
ii) Retained on 125 micrometre IS sieve, percent by mass, *Max*	10
iii) Retained on 63 micrometer IS sieve, percent by mass	50 *Min* 70 *Max*

Sieving Requirements	*Limit*
g) Size 150/90 micrometre A	
i) Retained on 150 micrometre IS sieve	Nil
ii) Retained on 90 micrometre IS sieve, percent, *Min*	50

(CHD 26)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

(First Revision)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

(First Revision)

FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 December 1981, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1967. The Sectional Committee decided to revise it in the light of experience gained since its publication. In the revised standard, the requirements of hygroscopicity and sulphur and their methods of tests have been added. Changes have been made in the calculation of sodium compounds and the procedure for determination of grit.

0.3 Barium nitrate is used in pyrotechnics, green flares, tracer bullets, primers and detonators. In small proportions, it is used as a source of barium oxide in enamels.

0.4 This standard contains clauses **2.2** and **3.1** which call for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for barium nitrate, intended primarily for use in pyrotechnics and explosives.

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*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — Barium nitrate shall be in the form of clean white crystals, free from foreign matter and visible impurities.

2.2 Particle Size — The particle size of the material shall be as agreed to between the purchaser and the supplier.

2.3 The material shall comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of **the** Table 1.

TABLE 1 REQUIREMENTS FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, *Max*	0.5	A-3
ii)	Matter insoluble in water, percent by mass, *Max*		
	a) Total	0.5	A-4
	b) Organic matter	0.1	
iii)	Hygroscopicity, percent by mass, *Max*	0.5	A-5
iv)	Grit, percent by mass, *Max*	0.05	A-6
v)	*p*H (of 5 percent solution)	5.5 to 7.5	A-7
vi)	Chlorides (as NaCl), percent by mass, *Max*	0.25	A-8
vii)	Chlorates	To pass test	A-9
viii)	Nitrites (as KNO_2), percent by mass, *Max*	0.03*	A-10
ix)	Sodium compounds (as $NaNO_2$), percent by mass, *Max*	0.5	A-11
x)	Ammonium compounds (as NH_3), percent by mass, *Max*	0.01	A-12
xi)	Calcium compounds (as calcium nitrate), percent by mass, *Max*	0.05	A-13
xii)	Sulphur	Nil	A-14
xiii)	Barium nitrate content as $Ba(NO_3)_2$, percent by mass, *Min*	99.00	A-15

*For use in initiatory compositions, the nitrite content (as KNO_2) shall be not more than 0.005 percent by mass.

3. PACKING AND MARKING

3.1 Packing — Barium nitrate shall be supplied in sound, clean, dry packages, which shall ensure safe transportation by common carriers ana complete freedom from accidental contamination. The type and size of packages shall be subject to agreement between the purchaser and the supplier.

3.1.1 When the material is required to be transported by rail, the packing shall conform to the provisions of Indian Railways Conference Association, Red Tariff No. 18.

3.2 Marking — The packages shall be legibly marked with the following information:

a) Name of the material;

b) Net mass of contents;

c) Manufacturer's name and/or recognized trade-mark, if any;

d) Year of manufacture; and

e) Batch number to enable the lot of manufacture to be traced from records.

3.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may by granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material, number of tests to be performed on each and the criteria for acceptance shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3)

METHODS OF TEST FOR BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF SAMPLE

A-2.1 Mix the sample well by rotating the bottle several times and transfer a portion immediately into a wide-mouthed bottle and stopper it. Take care that no pieces of cork or sealing wax get mixed with the sample. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Use this *prepared sample* for test purposes.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 5 g of the *prepared sample* in a weighed, clean, dry squat form weighing bottle, and dry in an oven at 105 ± 2°C. Cool in a desiccator[1] and weigh till constant mass is obtained.

A-3.2 Calculation

$$\text{Moisture, percent by mass} = 100\frac{M_1}{M}$$

where

M_1 = loss in mass in g of the material after heating, and

M = mass in g of the material taken for the test.

A-4. MATTER INSOLUBLE IN WATER

A-4.1 Procedure — Take 5 g of the *prepared sample* in a 500-ml beaker and dissolve in about 100 ml of water. Filter the residue in a weighed Gooch crucible. Wash the residue with water till free from all soluble matter. Dry the crucible in an oven maintained at 105 ± 2°C. Cool in a desiccator and weigh till constant mass is obtained.

---

*Specification for water for general laboratory use (*second revision*).

A-4.1.1 Calculation

$$\text{Total matter insoluble in water, percent by mass} = \frac{(M_2 - M_1)}{M} \times 100$$

where

M_2 = mass in g of the residue and the crucible,

M_1 = tare in g of the empty crucible, and

M = mass in g of the sample taken for the test.

A-4.2 To find the percentage of organic matter insoluble in water, ignite the Gooch crucible with the residue in a muffle furnace at 800°C for half an hour. Cool and weigh till constant mass is obtained. The loss in mass is due to the burning of the organic matter.

A-4.2.1 Calculation

$$\text{Organic matter insoluble in water} = \frac{(M_2 - M_3)}{M} \times 100$$

where

M_2 = mass in g of the residue and crucible,
M_3 = mass in g of the ignited residue and the crucible, and
M = mass in g of the sample taken for the test.

A-5. DETERMINATION OF HYGROSCOPICTTY

A-5.1 Procedure — Weigh accurately about 5 g of the sample as dried in **A-3.1** in a dish of approximately 6 cm diameter and expose the dried material to an atmosphere of 90 percent relative humidity at 27 ± 1°C (*see* Note) for 24 h. Weigh again.

NOTE — The test atmosphere of 90 percent relative humidity at 27 ± 1°C may be obtained at follows:

Place in a desiccator an aqueous sludge of hydrated zinc sulphate or sodium chloride so that the area of the surface of the sludge is not less than 75 percent of the floor of the desiccator. Supports for keeping the sample in the desiccator shall be so arranged that there is minimum obstruction in the diffusion of water vapour within the desiccator and the height of the exposed sample above the sine sulphate sludge does not exceed 10 cm. The desiccator shall be maintained at a temperature of 27 ± 1°C.

A-5.2 Calculation

$$\text{Hygroscopicity, percent by mass} = 100\,\frac{M_2 - M_1}{M_1}$$

where

M_2 = mass in g of the material after exposure, and

$M1$ = mass in g of the prepared sample taken for the test.

A-6. DETERMINATION OF GRIT

A-6.0 General — Grit is that portion of the ignited residue which **is** insoluble in aqua regia and 50 percent sodium hydroxide solution and is retained on a 125-micron IS sieve and is capable of scratching soda glass.

A-6.1 Reagents

A-6.1.1 *Aqua Regia* — Prepared by mixing 3 volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

A-6.1.2 *Sodium Hydroxide Solution* — 50 percent (*m\v*).

A-6.1.3 *Dilute Hydrochloric Acid* — 2 percent (*v/v*).

A-6.2 Procedure — Dissolve about 25 g of the *prepared sample* in water. Filter through a Whatman filter paper No, 40, and wash thoroughly. Dry the paper and ignite gently. Boil the ignited residue in 10 ml of aqua regia. Dilute with water and decant. Treat the residue with 20 ml of sodium hydroxide solution. Then dilute immediately with 200 to 300 ml of water, wash the gritty matter three times with water by decantation and then once with dilute hydrochloric acid. Filter on Whatman filter paper No. 40. Wash, ignite gently, cool and weigh. Brush the ignited gritty matter on 125-micron IS sieve and weigh the portions retained. Carry out the soda glass scratching test as prescribed in **A-6.4.** If the residue scratches soda glass, express the mass (primarily detained) as percentage grit.

A-6.3 Calculation

$$\text{Grit, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the grit retained on the sieve, and

M_2 = mass in g of the material taken for the test.

A-6.4 Soda Glass Scratching Test — Take a portion of the residue, retained on 125-micron IS sieve, in between two clear glass slides and press the slides by band along the length and breadth of the glass.

Examine whether any scratch is produced in the glass. If there is a scratch in the glass, then the residue is termed as grit.

A-7. DETERMINATION OF *p*H

A-7.1 Procedure — Shake 5.0 g of the material (passing through 150-micron IS sieve) with 75 ml of freshly boiled and cooled water. Filter and wash the residue thoroughly with 25 ml of water. Determine the *p*H of the aqueous extract with a suitable *p*H meter. Make at least two determinations from two separate portions and if the value differs by more than 0.4, repeat with two fresh portions. Reject the highest and the lowest and report the average.

NOTE — Excessive contact with air shall be avoided.

A-8. DETERMINATION OF CHLORIDES

A-8.0 Any sulphide present is eliminated by boiling with nitric acid. Chloride is then precipitated by adding a known excess of standard silver nitrate solution. The excess of silver nitrate is determined by titrating with a standard alkali thiocyanate solution using a ferric salt solution as indicator. Excess of thiocyanate reacts with the ferric ions to give a reddish-brown colour indicating completion of the reaction.

A-8.1 Reagents

A-8.1.1 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-8.1.2 *Standard Silver Nitrate Solution* – 0.1 N.

A-8.1.3 *Standard Ammonium Thiocyanate Solution* — 0.1 N.

A-8.1.4 *Ferric Alum Indicator* — saturated solution in water.

A-8.1.5 *Nitrobenzene*

A-8.2 Procedure — Weigh 5 g of the *prepared sample* and dissolve in about 100 ml of water. Filter the solution through a No. 40 Whatman filter paper into a conical flask. Wash the residue using 5 to 10 ml of water. Add to the filtrate 10 ml of nitric acid and a few glass beads to avoid bumping of the solution during the subsequent boiling operation. Keep a funnel over the mouth of the flask, boil the solution for 30 min and filter. Add to the filtrate 25 ml of silver nitrate solution and 5 ml of nitrobenzene. Shake vigorously till spongy flakes of silver chloride **are** obtained. Add 5 ml of ferric alum indicator solution and titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change. Run a blank simultaneously.

---

*Specification for nitric acid (*first revision*).

A-8.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{5.846 (V_1 - V_2) N}{M}$$

where

V_1 = volume in ml of ammonium thiocyanate solution used in the blank,

V_2 = volume in ml of ammonium thiocyanate solution used for the sample,

N = normality of ammonium thiocyanate solution, and

M = mass in g of the *prepared sample* taken for the test.

A-9. TEST FOR CHLORATES

A-9.1 Reagents

A-9.1.1 *Aniline Hydrochloride* — Dissolve 18 g of redistilled aniline in in 375 ml of concentrated hydrochloric acid and make up the volume to 500 ml with water. Add two drops of saturated potassium chlorate solution, shake and allow to stand overnight. Filter off the blue sediment and store the reagent in a glass stoppered bottle in the dark.

A-9.2 Procedure — Dissolve 5 g of the *prepared sample* in 40 ml of warm water, filter if necessary, and make up to 100 ml with aniline hydrochloride reagent.

A-9.2.1 The material shall be considered to have passed the test if no blue colouration is developed in 10 min.

A-10. DETERMINATION OF NITRITES

A-10.0 Principle — Nitrites are determined colorimetrically by visual comparison using dimethylaniline hydrochloride.

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50-ml capacity

A-10.2 Reagents

A-10.2.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-10.2.2 *Dimethylaniline Hydrochloride* — Dissolve 10 g of dimethylaniline in 100 ml of hydrochloric acid.

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*Specification for hydrochloric acid (*second recision*).

A-10.2.3 *Standard Potassium Nitrite Solution* — Dissolve 1.0 g of potassium nitrite in water, and make up to 1 000 ml. One millilitre of the solution is equivalent to 0.001 g of nitrite (as KNO_2).

A-10.3 Procedure — Dissolve 10.0 g of the *prepared sample* in 25 ml of water and filter, if necessary. Wash the filter paper and transfer both the filtrate and washings into a Nessler cylinder. Add four drops of concentrated hydrochloric acid and 0.5 ml of dimethylaniline hydrochloride solution and make up the volume to 50 ml. Carry out a control test in another Nessler cylinder using 3 ml of standard potassium nitrite solution in place of the material and the same quantities of the other reagents. Compare the colour after 20 min.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-11. DETERMINATION OF SODIUM COMPOUNDS

A-11.0 Qualitative Test for Sodium — Dissolve 5 g of the *prepared sample* in 23-cm of water and acidify with concentrated hydrochloric acid (*see* IS : 265-1976*). Dip a clean platinum wire into the edge of a colourless Barisen flame. When it is found to be colourless, remove from the flame, cool and dip it in the solution of the sample. The presence of sodium radical is established if the characteristic yellow colour is imparted to the flame. If sodium is present, estimate it as prescribed in **A-11.2** or **A-11.3.**

A-11.1 General — Two methods are pcescribed for the determination of sodium, namely, gravimetric method and photometric method. In case of dispute, photometric method shall be the referee method.

A-11.2 Gravimetric Method

A-11.2.1 *Principle* — Barium is separated from sodium and calcium by precipitation as sulphate and the filtrate containing sodium and calcium sulphates is evaporated to dryness and ignited to remove free sulphuric acid.

A-11.2.2 *Procedure* — Dissolve 5 g of the *prepared sample* in 200 ml of water, heat until boiling and add drop by drop 50 ml of 10 percent sulphuric acid. Continue boiling for about 20 min and allow to stand overnight. Then filter and wash the precipitate. Evaporate the filtrate and washings to dryness in a tared silica dish and ignite it at a dull red heat to remove sulphuric acid. Cool in a desiccator and weigh.

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*Specific for hydrchloric acid (*second revision*).

A-11.2.3 *Calculation* — From the mass of the residue of sodium and calcium sulphates deduct the mass of calcium sulphate calculated from **A-13.** Express the balance mass as sodium nitrate and record as percentage of the original sample.

A-11.3 Flame Photometer Method

A-11.3.0 *General* — Sodium content is determined with the help of a flame emission spectrophotometer by measuring the intensity of radiation emitted at 589 nm. For this purpose, use may also be made of a filter type flame photometer. The intensity of radiation either at the specified wavelength, or through the relevant filter, is compared with that obtained for simulated standard solutions prepared from standard sodium chloride solution.

NOTE — Water used in preparing standard solutions shall be double-distilled so that it produces no deflections of the galvanometer when the instrument is set for full scale deflection for 5 ppm of sodium concentration.

A-11.3.1 *Apparatus*

A-11.3.1.1 *Flame photometer* — provided with arrangement for monochromatic light or a set of filters required for determination of sodium (*see* **A-11.3.0**). The photometer comprises essentially a galvanometer, a photo cell or tube and a photo-multiplier tube. Other accessories are an atomizer-burner (or an atomizer and a burner) fed with town gas or liquified petroleum gas (LPG) and compressed air (or oxygen, acetylene, etc) in such a ratio that combustion of gas is complete.

A-11.3.1.2 *Burette* — of 25-ml nominal capacity and graduated in 0.05 ml for class A accuracy (*see* IS : 1997-1967*).

A-11.3.1.3 *Volumetric flasks* — 1 000-ml, 250-ml and 100-ml nominal capacity of Class A accuracy (*see* IS : 915-1975†).

A-11.3.2 *Reagents*

A-11.3.2.1 *Dilute hydrochloric acid* 1: 1 (*v/v).*

A-11.3.2.2 *Standard sodium solution* — Dissolve 2.543 g of sodium chloride in water in 1 000-ml volumetric flask and dilute to the mark. Shake well. This solution contains 1 mg of Na/ml of the solution.

To prepare 1, 2, 3, 4, 5 ppm of sodium solution, transfer 1, 2, 3, 4, 5 ml each of stock solution in five different 1 000-ml flasks by means of a pipette and dilute up to the mark with water. Shake well. Mark them as 1, 2, 3, 4, 5 ppm of sodium solution.

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*Specification for burettes *(first revision).*

†Specification for one-mark volumetric flasks *(first revision).*

A-11.3.2.3 *Standard potassium solutions* — Weigh accurately 0.381 4 g of potassium chloride (KCl), previously dried at 110 ± 2°C to constant mass. Dissolve in water, transfer to a 100-ml volumetric flask, dilute up to the mark with water and mix.

A-11.3.3 *Setting up the Apparatus* — Switch the instrument on and wait for its stabilization. Adjust the slit and wavelengths at 589 nm or place the filters in position as the case may be.

A-11.3.3.1 Pour water in the spray cup, open its tap and turn the compressed air or oxygen on. Adjust the tap and pressure to establish atomization of water in a fine spray. Open the gas tap and light the burner. Adjust gas, if necessary, to get complete combustion. Next switch the photocell on and adjust the indicator needle or the indicator light spot, as the case may, to bring it to the 'zero' position.

A-11.3.3.2 Atomize sodium solution 5 ppm (**A-11.3.2.2**) in place of water (**A-11.3.3.1**) and adjust the deflection to full scale. Atomize water once again to check that the indicator needle or light spot rests at 'zero'. If it shifts from 'zero', again adjust it to 'zero'. Thus, when the instrument reads zero with water and shows full scale deflection for 5 ppm Na solution, it is ready for use.

NOTE 1 — Atomization of any solution should always be followed by atomization of double-distilled water (*see* Note in **A-11.3.0**) till the indicator conies to zero position.

NOTE 2 – DO not disturb the setting of the atomizer or burner so that the rates of flow are constant for a series of determinations.

A-11.3.4 *Procedure*

A-11.3.4.1 *Preparation of the sample solution* — Weigh accurately 0.5 g of the sample previously dried at 110 ± 2°C to constant mass. Dissolve in water, transfer to a 250-ml volumetric flask, dilute up to the mark with water and mix.

A-11.3.4.2 *Calibration graph and determination* — Plot a calibration graph using the above known solutions of sodium. Then atomize the sample solution. Note the deflection and find out the reading from the graph which would give directly ppm of sodium.

Calculation — Sodium compounds (as Na), percent by mass

$$\frac{X}{10^6} \times \frac{250}{M} \times \frac{100}{1}$$

Sodium compounds (as Na No_3), percent by mass $= P \times 3.7$

where

X = reading of Na from the graph,
M = mass in g of the material taken for the test, and
P = percentage of sodium determined.

A-12. DETERMINATION OF AMMONIUM COMPOUNDS

A-12.0 Principle — Nessler reagent (K_2HgI_4) reacts with ammonia to form a reddish-brown colloidal compound with the empirical formula $NH_2Hg_2I_3$.

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* — 50-ml capacity,

A-12.2 Reagents

A-12.2.1 *Sodium Hydroxide Solution* — approximately 5 N.

A-12.2.2 *Nessler Solution* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly with stirring saturated mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution, and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

A-12.2.3 *Standard Ammonium Chloride Solution* — Dissolve 0.314 1 g of ammonium chloride in water and make up to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of ammonia (as NH_3).

A-12.3 Procedure — Weigh accurately l.00 g of the *prepared sample* and dissolve in 20 ml of water in a Nessler cylinder. Add 5 ml of sodium hydroxide solution and 1 ml of Nessler solution. Make up the solution to 50 ml. Carry out a control test in another Nessler cylinder with 1 ml of standard ammonium chloride solution in place of the material and the same quantities of reagents. Compare the colour produced.

A-12.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-13. DETERMINATION OF CALCIUM COMPOUNDS

A-13.0 Principle — Calcium picrolonate, when dissolved in hot water, develops a pink to red colour with bromine and sodium hydroxide.

A-13.1 Reagents

A-13.1.1 *Picrolonic Acid* — 3-Methyl-4-nitro - 1 - (*p*-nitrophenyl) - 5 - pyrazolone.

A-13.1.2 *Bromine Water* — saturated.

A-13.1.3 *Ether* — *see* IS : 336-1973*.

A-13.1.4 *Rectified Spirit* – *see* IS : 323-1959†.

A-13.1.5 *Sodium Hydroxide Solution* — 2 N (approximately).

A-13.2 Procedure — Dissolve the residue of sodium and calcium sulphates obtained in **A-11,** in water. Warm the solution and add 2 g of picrolonic acid dissolved in 750 ml of warm water. After allowing to stand for several hours, collect the precipitate on a sintered glass crucible G3 and, after washing with cold water, wash with diethylether. Dissolve in hot water and add 1 mi of bromine water for every 50 ml of calcium picrolonate solution. After heating the mixture on a water-bath for 10 min, add 10 ml of rectified spirit and after allowing to cool, introduce 2 ml of sodium hydroxide solution. The pink colour thus produced is compared with those of standards similarly prepared, starting with known amounts of calcium.

A-14. TEST FOR SULPHUR

A-14.0 Outline of the Method — The yellow alkaline solution of sodium nitroprusside [Na_2. Fe (CN $)_5$ NO] gives a red/violet colour with soluble sulphide.

A-14.1 Reagent

A-14.1.1 *Sodium Nitroprusside Solution* — one percent (*m/v*), made slightly alkaline with ammonium hydroxide.

A-14.2 Procedure — Dissolve about 1 g of the *prepared sample* in 100 ml of water and make it just alkaline with dilute ammonium hydroxide. Mix a drop of the solution on a spot plate with freshly prepared solution of sodium nitroprusside. Alternatively, the test may be carried out on a filter paper impregnated with 5 percent solution of ammoniacal sodium nitroprusside.

A-14.2.1 The material shall satisfy the requirement prescribed in Table 1 if there is no red/violet colouration.

A-15. DETERMINATION OF BARIUM NITRATE CONTENT

A-15.0 Principle — Barium nitrate is determined by precipitating with dilute sulphuric acid as barium sulphate, igniting the precipitate and calculating as barium nitrate.

Specification far ether *(second revision*).

†Specification for rectified spirit (*revised*).

A-15.1 Reagents

A-15.1.1 *Dilute Hydrochloric Acid* — 5 N.

A-15.1.2 *Dilute Sulphuric Acid* — approximately 1 : 2 and 1 : 99.

A-15.2 Procedure — Dissolve about 0.5 g of the *prepared sample,* accurately weighed in about 20 ml of water and 1 ml of dilute hydrochloric acid. Heat the solution to boiling, add 5 ml of hot dilute sulphuric acid (1 : 2) slowly with constant stirring. Cool, dilute to about 100 ml and allow to stand for 4 h at 60°C. Filter the precipitate through filter paper (Whatman No. 42 or equivalent). Wash the precipitate twice with dilute sulphuric acid (1 : 99) and then with hot water till it is free from chloride and sulphate. Dry the precipitate and then ignite slowly at a low flame and finally at 800°C in a furnace till constant mass is obtained.

A-15.2.1 *Calculation*

$$\text{Barium nitrate [As Ba (NO}_3\text{)}_2\text{]} = 112 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the ignited residue, and
M = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF BARIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling **test samples, the** following precautions and directions shall be observed.

B-1.1 Samples shall not be drawn in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* **3.2**) and the date of sampling.

B-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of the material all the containers of the same type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

B-2.1.l For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE N (1)			SAMPLE SIZE n (2)
Up	to	15	3
16	”	25	4
26	”	50	5
51	”	100	7
101 and above			10

B-2.1.2 In order to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure is recommended for use:

> Arrange the containers in the lot in a systematic order and starting from any one, count them as 1, 2, 3, ... etc, up to r and so on, where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of the material not less than 100 g in mass.

B-3.2 Out of these portions, a small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of mass not less than 150 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts and transferred to separate bottles. The material in each bottle constitutes an individual sample. One of these sets (each set containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.4 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of moisture and matter insoluble in water shall be performed on each of the individual samples.

B-4.2 Tests for determination of all the remaining characteristics given in Table 1 shall be carried out on the composite sample.

B 5. CRITERIA FOR CONFORMITY

B-5.1 From each set of the individual test results for moisture and matter insoluble in water, the mean ($\bar{X}$) and range (R) of test results shall be computed separately (the range being defined as the difference between the maximum and the minimum values of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of moisture and matter insoluble in water, if ($\bar{X} + 0.6R$) as calculated from the relevant test results is less than or equal to the relevant maximum value specified in Sl No. 1 and 2 of Table 1.

B-5.2 For declaring the conformity of the lot to the specification requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.S
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertx	Hz	1 Hz = 1 c/s (s^{-2})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V - 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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